Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*Z*)-2-Anilino-2-[oxido(phenyl)iminio]-*N*-(2-pyridyl)acetamide methanol 0.425-solvate

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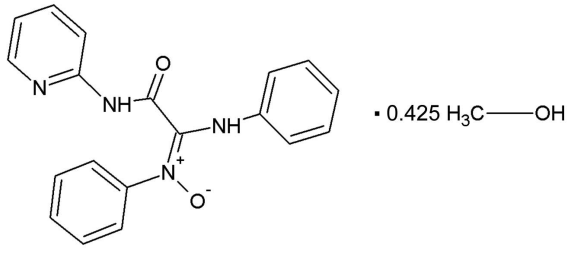
Received 24 September 2007; accepted 30 January 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.067; wR factor = 0.156; data-to-parameter ratio = 14.1.

The title compound, $C_{19}H_{16}N_4O_2\cdot0.425CH_4O$, crystallizes with two formula units per asymmetric unit. Researching its crystal structure constitutes part of a study of the nature of interactions between the N^+-O^- group and the vicinal NH group. The nitrone group and methanol solvent molecules are linked *via* four N—H···O and one O—H···O hydrogen bonds, with donor–acceptor distances of 2.603 (3)–2.730 (3) and 2.770 (3) Å, respectively. The crystal structure also involves two intermolecular N—H···N hydrogen bonds.

Related literature

For literature related to the synthesis of the title compound, see: Branco *et al.* (1992); Aurich (1968); Guimanini *et al.* (1999); Szantay *et al.* (1965); Southan *et al.* (1998); Warshaw *et al.* (1989). For literature on nitrones as an active equivalent of the C=O group, see: Boruah *et al.* (2003); Freisleben *et al.* (2002); Saito *et al.* (2001); Gravestock *et al.* (2000); Torssell (1988). For examples of nitrones with a vicinal NH group, see: Janzen *et al.* (1997); Clement *et al.* (1987); Baranowska *et al.* (1977); Aurich *et al.* (1976); Rosenberg *et al.* (1972). For literature on the medical use of these compounds, see: Floyd (2006). For related literature, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{19}H_{16}N_4O_2\cdot0.425CH_4O$
M_r = 345.98
Monoclinic, $P2_1/c$
a = 18.7604 (3) Å
b = 9.4701 (2) Å
c = 21.2839 (5) Å
β = 104.375 (1)°
V = 3662.97 (13) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.11 × 0.09 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.991, T_{max} = 0.998
12501 measured reflections
6668 independent reflections
3520 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.156
S = 1.03
6668 reflections
474 parameters
548 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N7*A*—H7*A*···O13*B*	0.86	1.94	2.730 (3)	152
N11*A*—H11*A*···N1*B*^{i}	0.86	2.30	3.095 (3)	153
N11*A*—H11*A*···O13*A*	0.86	2.25	2.603 (3)	105
N7*B*—H7*B*···O13*A*^{ii}	0.86	1.90	2.724 (3)	160
N11*B*—H11*B*···N1*A*	0.86	2.32	3.056 (3)	144
N11*B*—H11*B*···O13*B*	0.86	2.28	2.604 (3)	102
O51—H51···O13*B*	0.82	1.95	2.770 (3)	179

Symmetry codes: (i) $x, y+1, z$; (ii) $x, y-1, z$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

The authors thank the Joint X-ray Laboratory, Faculty of Chemistry, and SLAFiBS, Jagiellonian University, for making available the Nonius KappaCCD diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2043).

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supporting information

Acta Cryst. (2008). E**64**, o599–o600 [doi:10.1107/S1600536808003346]

(2*Z*)-2-Anilino-2-[oxido(phenyl)iminio]-*N*-(2-pyridyl)acetamide methanol 0.425-solvate

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S1. Comment

Nitrones have attracted attention of organic chemists as nitrone N^+–O^- moiety can be treated as an active equivalent of C=O group (Boruah *et al.* 2003, Freisleben *et al.* 2002, Saito *et al.* 2001, Gravestock *et al.* 2000, Torssell 1988, Floyd 2006). However, only several examples of nitrones bearing a vicinal NH group have been reported so far (Janzen *et al.* 1997, Clement *et al.* 1987, Baranowska *et al.* 1977, Aurich *et al.* 1976, Rosenberg *et al.* 1972). As reported, the compounds could be derived from nitriles (Branco *et al.* 1992), imidoformic acid esters or *α*-chloroimines (Aurich 1968), hydroxylamines and methylene amines (Aurich 1968, Guimanini *et al.* 1999), secondary amines (Szantay *et al.* 1965), and nitroso compounds, from hydroxyguanidines (Southan *et al.* 1998) or from other nitrones (Warshaw *et al.*1989). We have succeeded in developing a straightforward way of obtaining nitrones with a vicinal NH group in excellent yields, starting from easily available pyridilides of 3-oxobutanoic acid and nitrosobenzene. The crystal structure analysis of (2*Z*)-2-anilino-2-[oxido(phenyl)imino]-*N*-pyridin-2-ylacetamide was performed in order to determine the nature of the interactions between the N^+–O^- moiety of the title nitrone with the vicinal NH group; this should help us to understand the compound's versatile reactivity towards various diamines. The symmetrically independent part of the unit cell is composed of two 2-anilino-2-[oxido(phenyl)imino]-*N*-pyridin-2-ylacetamide molecules and a 0.85 methanol molecule disordered between two positions with partial occupancy parameters of 0.586 (7) and 0.264 (7) for O51A—C51A and O51B—C51B, respectively. The conformation of the two symetrically independent nitrone molecules is shown in Figs. 1a and 1 b. No appreciable differences can be observed between the bond lengths and angles of the independent nitrone molecules and they are comparable with the values reported in the literature (Allen *et al.* 1987). The planarity of the C–C=N^+→(O^-) part with *Z* configuration, in respect of N12–C10 double bond, observed for both symmetrically independent molecules is the most interesting feature of the investigated molecules, from the geometrical point of view. The O13—N12—C10—N11 and O13—N12—C10—C8 torsion angles are 2.6° and -174.3°, respectively for molecule A and -4.1° and 174.4°, respectively for molecule B. Significant torsion angles observed for nitrone molecules A and B are compared in Table 1. A l l aryl rings are twisted against each other: ⟨ (C14···C19/N1···C6) = 68.0 (1)° for A and 53.8 (1)° for B; ⟨ (C20···C25/N1···C6) = 75.5 (1)° for A and 66.4 (1)° for B. The crystal packing (Fig. 2) is controlled by N—H···O, N—H···N and O—H···O hydrogen bonds and weak van der Waals interactions (Table 2). The two N atoms (N7 and N11) in each molecule of the title compound are involved in the hydrogen bonds as donors forming two intra- and one intermolecular hydrogen bonds (Table 2). The intermolecular N11—H11···N1 interactions are relatively weak, with donor–acceptor distances of 3.095 (3) Å and 3.056 (3) Å due to simultaneous donor participation in intramolecular N11—H11···O13 interactions.

S2. Experimental

Slow recrystallization from methanol at room temperature afforded crystals suitable for X-ray measurements. The ratio (2*Z*)-2-anilino-2-[oxido(phenyl)imino]-*N*-pyridin-2-ylacetamide to metanol molecule equals 2: 0.85 was confirmed by elemental analysis.

S3. Refinement

The contents of methanol in the asymmetric unit was determined by the refinement of structural parametres assuming its site occupancy factor in the range 1.000 - 0.500. The best solution (using wR2 as criterion) was found for 0.850 methanol molecule per two nitrone molecules. All H atom positions were observed in difference Fourier map. Nevertheless, in the refinement procedure the hydrogen atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å for aromatic CH, N—H = 0.86 Å. In methanol molecule CH_3 group with assumed tetrahedral angles was refined including free rotation about the C—O bond, C—H = 0.96 Å and O—H = 0.82 Å. U_{iso}(H) = 1.5U_{eq}(C) for methyl groups in methanol and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

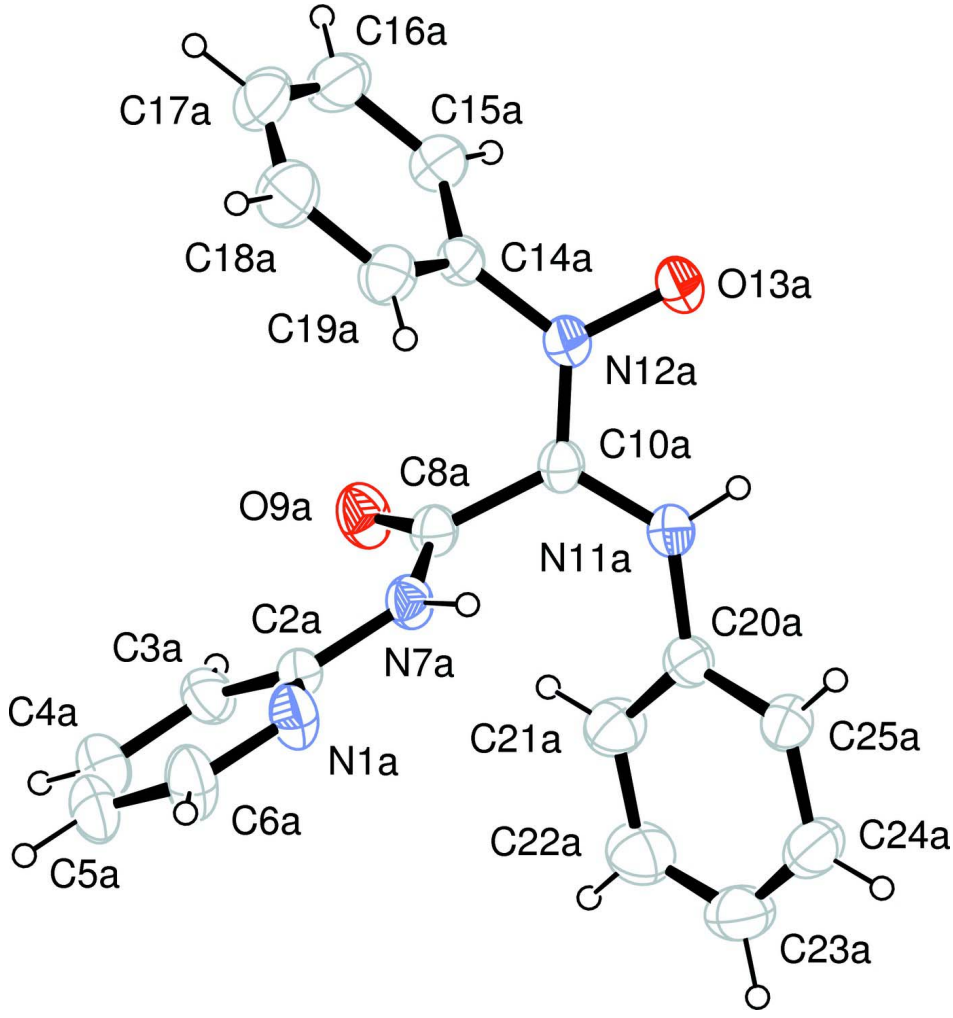

Figure 1

An *ORTEP-3* (Farrugia, 1997) view of the nitrone molecules with the crystallographic numbering scheme of atoms: the conformation of A molecule in projecton on C8AN11AN12A plane. Displacement ellipsoids of non-H atoms are drawn at 30% probability level.

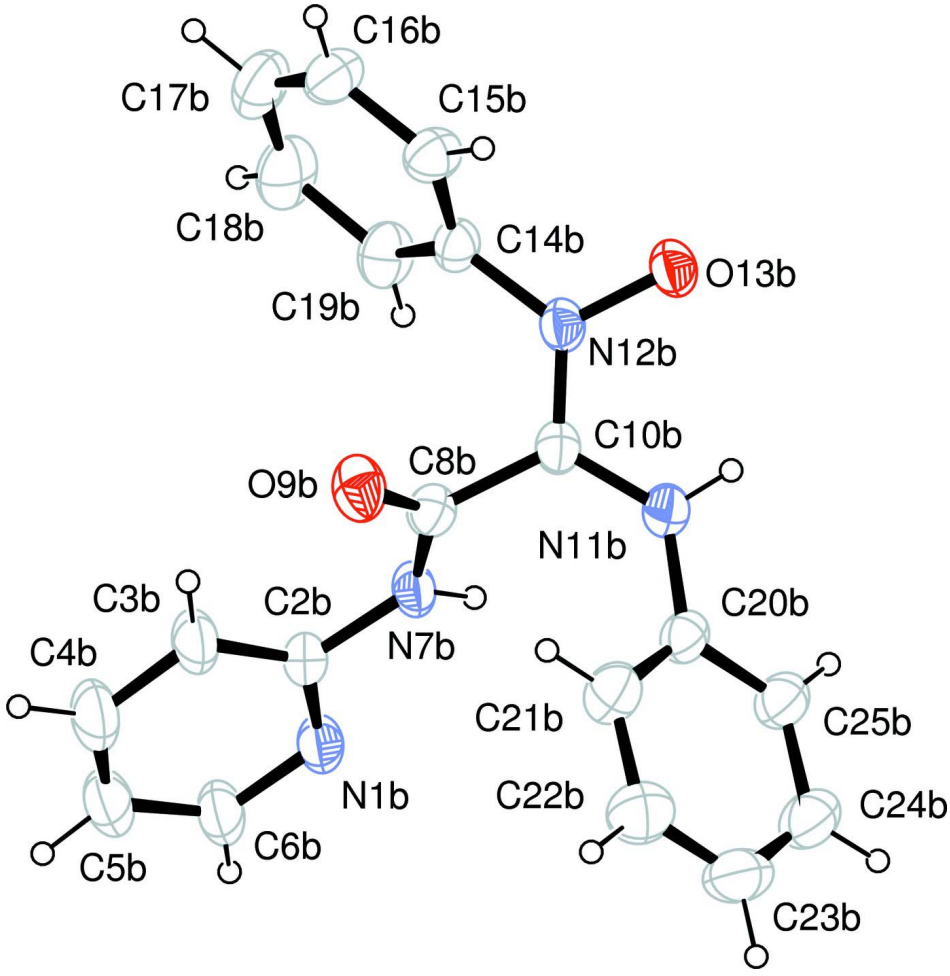

Figure 2

An *ORTEP-3* (Farrugia, 1997) view of the nitrone molecules with the crystallographic numbering scheme of atoms: the conformation of B molecule in projecton on C8BN11BN12B plane; Displacement ellipsoids of non-H atoms are drawn at 30% probability level.

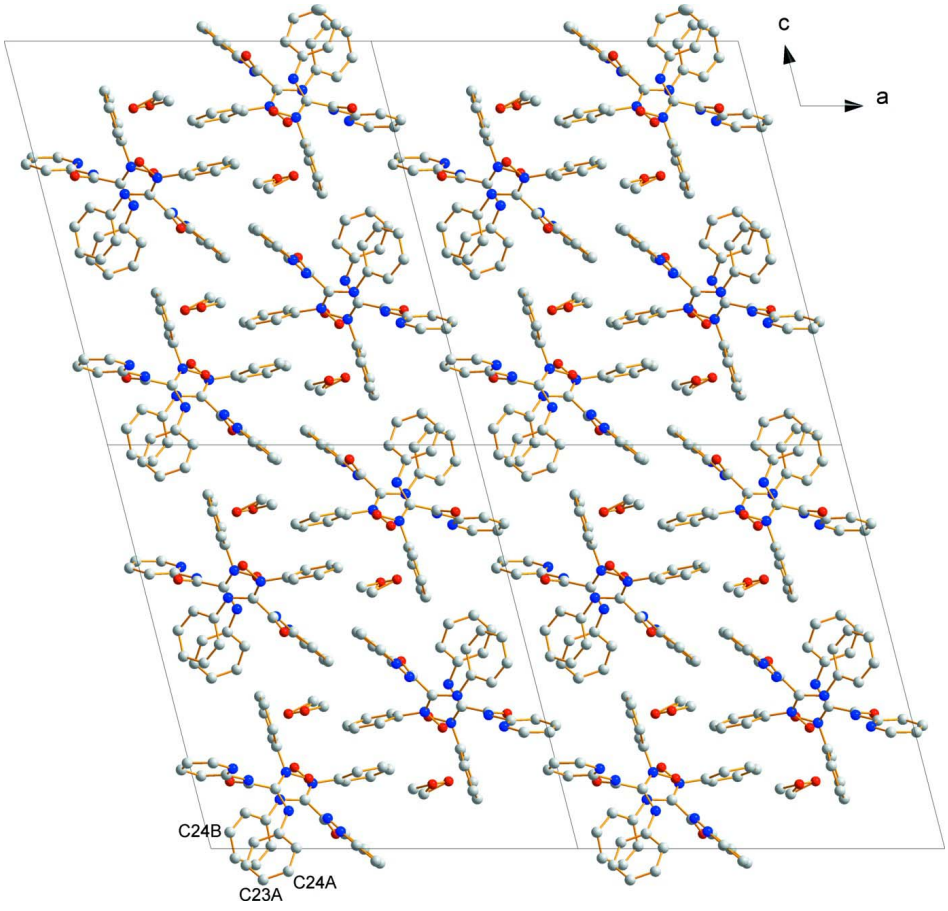

Figure 3

Drawing of the crystal packing viewed along [010] (Brandenburg, 2006). Hydrogen atoms were omitted for clarity.

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Crystal data

$C_{19}H_{16}N_4O_2 \cdot 0.425CH_4O$
$M_r = 345.98$
Monoclinic, $P2_1/c$
$a = 18.7604$ (3) Å
$b = 9.4701$ (2) Å
$c = 21.2839$ (5) Å
$\beta = 104.375$ (1)°
$V = 3662.97$ (13) Å^3
$Z = 8$
$F(000) = 1453$
$D_x = 1.255$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6743 reflections
$\theta = 1.0$–$25.4°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Plate, colourless
0.11 × 0.09 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Horizontally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans to fill asymmetric unit
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.991$, $T_{max} = 0.998$
12501 measured reflections
6668 independent reflections
3520 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$

θ_{max} = 25.4°, θ_{min} = 3.4°
h = 0→22
k = −10→11
l = −25→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.156
S = 1.03
6668 reflections
474 parameters
548 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0657P)^2 + 0.3423P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0055 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1A	0.11545 (13)	0.6719 (2)	0.19762 (11)	0.0661 (6)	
C2A	0.09371 (14)	0.8040 (3)	0.18252 (11)	0.0483 (6)	
C3A	0.02414 (14)	0.8515 (3)	0.17983 (13)	0.0616 (7)	
H3A	0.0107	0.9443	0.1682	0.074*	
C4A	−0.02552 (16)	0.7579 (4)	0.19488 (15)	0.0771 (9)	
H4A	−0.0732	0.7871	0.1935	0.092*	
C5A	−0.00442 (19)	0.6229 (4)	0.21166 (16)	0.0817 (9)	
H5A	−0.0368	0.5585	0.2226	0.098*	
C6A	0.06588 (19)	0.5847 (3)	0.21201 (16)	0.0828 (10)	
H6A	0.0801	0.4920	0.2230	0.099*	
N7A	0.14984 (11)	0.8889 (2)	0.16925 (9)	0.0501 (6)	
H7A	0.1889	0.8460	0.1652	0.060*	
C8A	0.14887 (14)	1.0299 (3)	0.16219 (12)	0.0502 (7)	
O9A	0.09855 (10)	1.10851 (19)	0.16598 (9)	0.0668 (5)	
C10A	0.21768 (14)	1.0904 (3)	0.14793 (13)	0.0471 (6)	
N11A	0.23810 (11)	1.0608 (2)	0.09302 (10)	0.0524 (6)	
H11A	0.2816	1.0874	0.0919	0.063*	
N12A	0.25797 (11)	1.1807 (2)	0.18870 (10)	0.0499 (5)	
O13A	0.31518 (9)	1.24341 (18)	0.17334 (8)	0.0604 (5)	
C14A	0.25051 (13)	1.2046 (3)	0.25341 (12)	0.0504 (7)	
C15A	0.24581 (15)	1.3418 (3)	0.27317 (14)	0.0670 (8)	

H15A	0.2466	1.4164	0.2449	0.080*
C16A	0.23996 (17)	1.3673 (4)	0.33547 (17)	0.0835 (9)
H16A	0.2365	1.4596	0.3494	0.100*
C17A	0.23921 (18)	1.2574 (5)	0.37671 (17)	0.0926 (11)
H17A	0.2343	1.2749	0.4184	0.111*
C18A	0.24558 (19)	1.1217 (4)	0.35712 (16)	0.0885 (10)
H18A	0.2458	1.0474	0.3858	0.106*
C19A	0.25170 (16)	1.0944 (3)	0.29510 (14)	0.0693 (8)
H19A	0.2566	1.0022	0.2818	0.083*
C20A	0.19658 (15)	0.9913 (3)	0.03686 (12)	0.0496 (7)
C21A	0.12171 (17)	1.0074 (3)	0.01520 (14)	0.0697 (8)
H21A	0.0962	1.0617	0.0388	0.084*
C22A	0.0844 (2)	0.9430 (4)	−0.04161 (17)	0.0858 (10)
H22A	0.0335	0.9522	−0.0554	0.103*
C23A	0.1207 (2)	0.8662 (4)	−0.07771 (16)	0.0854 (10)
H23A	0.0951	0.8253	−0.1165	0.102*
C24A	0.1954 (2)	0.8495 (3)	−0.05644 (16)	0.0801 (9)
H24A	0.2208	0.7974	−0.0810	0.096*
C25A	0.23329 (16)	0.9101 (3)	0.00149 (14)	0.0619 (7)
H25A	0.2837	0.8958	0.0165	0.074*
N1B	0.36559 (12)	0.1853 (2)	0.04201 (11)	0.0607 (6)
C2B	0.37519 (14)	0.3237 (3)	0.03646 (13)	0.0536 (7)
C3B	0.41689 (16)	0.3812 (3)	−0.00164 (15)	0.0749 (9)
H3B	0.4225	0.4785	−0.0042	0.090*
C4B	0.45036 (19)	0.2895 (4)	−0.03606 (17)	0.0926 (11)
H4B	0.4791	0.3245	−0.0624	0.111*
C5B	0.44097 (18)	0.1478 (4)	−0.03111 (17)	0.0851 (10)
H5B	0.4628	0.0846	−0.0541	0.102*
C6B	0.39919 (17)	0.1009 (3)	0.00806 (16)	0.0772 (9)
H6B	0.3935	0.0038	0.0116	0.093*
N7B	0.33914 (11)	0.4052 (2)	0.07475 (10)	0.0536 (6)
H7B	0.3210	0.3604	0.1023	0.064*
C8B	0.33054 (14)	0.5460 (3)	0.07221 (13)	0.0532 (7)
O9B	0.35086 (11)	0.62721 (19)	0.03605 (9)	0.0702 (6)
C10B	0.28999 (14)	0.6032 (3)	0.12011 (12)	0.0485 (7)
N11B	0.22196 (12)	0.5613 (2)	0.12126 (11)	0.0564 (6)
H11B	0.2086	0.5778	0.1564	0.068*
N12B	0.32199 (11)	0.7001 (2)	0.16157 (10)	0.0517 (6)
O13B	0.28421 (9)	0.76002 (18)	0.20063 (8)	0.0605 (5)
C14B	0.39782 (14)	0.7415 (3)	0.17218 (12)	0.0517 (7)
C15B	0.41339 (16)	0.8830 (3)	0.16710 (13)	0.0647 (8)
H15B	0.3756	0.9489	0.1561	0.078*
C16B	0.48549 (18)	0.9248 (4)	0.17850 (14)	0.0761 (9)
H16B	0.4965	1.0195	0.1743	0.091*
C17B	0.54132 (18)	0.8285 (5)	0.19598 (16)	0.0879 (10)
H17B	0.5901	0.8578	0.2038	0.105*
C18B	0.52519 (17)	0.6890 (4)	0.20198 (16)	0.0870 (10)
H18B	0.5632	0.6238	0.2143	0.104*

C19B	0.45248 (16)	0.6438 (3)	0.18974 (14)	0.0700 (8)	
H19B	0.4414	0.5489	0.1935	0.084*	
C25B	0.11891 (15)	0.4058 (3)	0.08793 (15)	0.0664 (8)	
H25B	0.1223	0.3862	0.1314	0.080*	
C24B	0.06318 (18)	0.3478 (3)	0.0403 (2)	0.0829 (10)	
H24B	0.0276	0.2922	0.0518	0.099*	
C23B	0.05954 (19)	0.3712 (3)	−0.0239 (2)	0.0888 (10)	
H23B	0.0224	0.3300	−0.0559	0.107*	
C22B	0.1108 (2)	0.4551 (4)	−0.04032 (18)	0.0916 (10)	
H22B	0.1086	0.4700	−0.0839	0.110*	
C21B	0.16563 (10)	0.51847 (18)	0.00578 (9)	0.0730 (8)	
H21B	0.1996	0.5776	−0.0062	0.088*	
C20B	0.16951 (10)	0.49270 (18)	0.07069 (9)	0.0549 (7)	
O51	0.30526 (10)	0.73186 (18)	0.33354 (9)	0.119 (2)	0.586 (7)
H51	0.2985	0.7400	0.2941	0.178*	0.586 (7)
C51	0.38176 (10)	0.76316 (18)	0.36484 (9)	0.133 (4)	0.586 (7)
H51A	0.3863	0.8606	0.3777	0.200*	0.586 (7)
H51B	0.4117	0.7453	0.3351	0.200*	0.586 (7)
H51C	0.3978	0.7043	0.4024	0.200*	0.586 (7)
O52	0.35159 (10)	0.68397 (18)	0.34082 (9)	0.143 (7)	0.264 (7)
H52	0.3218	0.6949	0.3058	0.215*	0.264 (7)
C52	0.40649 (10)	0.80407 (18)	0.35212 (9)	0.171 (11)	0.264 (7)
H52A	0.3823	0.8894	0.3339	0.257*	0.264 (7)
H52B	0.4455	0.7825	0.3318	0.257*	0.264 (7)
H52C	0.4265	0.8165	0.3979	0.257*	0.264 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0653 (16)	0.0595 (15)	0.0838 (17)	−0.0015 (12)	0.0379 (13)	0.0106 (13)
C2A	0.0439 (16)	0.0569 (17)	0.0461 (15)	−0.0054 (13)	0.0151 (13)	−0.0046 (13)
C3A	0.0460 (17)	0.0653 (18)	0.074 (2)	−0.0036 (14)	0.0162 (15)	−0.0060 (15)
C4A	0.0511 (19)	0.093 (2)	0.092 (2)	−0.0105 (17)	0.0272 (17)	−0.0084 (19)
C5A	0.079 (2)	0.087 (2)	0.092 (2)	−0.0212 (19)	0.046 (2)	0.0020 (19)
C6A	0.084 (2)	0.073 (2)	0.106 (3)	−0.0031 (18)	0.051 (2)	0.0174 (18)
N7A	0.0426 (12)	0.0511 (14)	0.0604 (14)	0.0030 (10)	0.0201 (11)	−0.0014 (10)
C8A	0.0447 (17)	0.0561 (19)	0.0515 (16)	0.0012 (14)	0.0154 (13)	−0.0050 (13)
O9A	0.0513 (12)	0.0618 (12)	0.0934 (15)	0.0078 (10)	0.0297 (11)	0.0021 (10)
C10A	0.0442 (15)	0.0483 (15)	0.0520 (17)	0.0052 (13)	0.0180 (14)	0.0013 (13)
N11A	0.0459 (13)	0.0593 (13)	0.0557 (14)	−0.0028 (11)	0.0192 (12)	−0.0051 (11)
N12A	0.0442 (13)	0.0550 (13)	0.0519 (14)	−0.0028 (11)	0.0150 (11)	−0.0001 (11)
O13A	0.0474 (11)	0.0753 (12)	0.0620 (12)	−0.0126 (9)	0.0201 (9)	−0.0008 (9)
C14A	0.0388 (15)	0.0658 (18)	0.0468 (16)	−0.0034 (13)	0.0109 (12)	−0.0041 (14)
C15A	0.067 (2)	0.071 (2)	0.064 (2)	−0.0014 (15)	0.0177 (16)	−0.0080 (15)
C16A	0.077 (2)	0.100 (2)	0.074 (2)	−0.0006 (19)	0.0199 (19)	−0.026 (2)
C17A	0.078 (2)	0.148 (3)	0.053 (2)	−0.011 (2)	0.0190 (18)	−0.020 (2)
C18A	0.095 (3)	0.112 (3)	0.057 (2)	−0.012 (2)	0.0167 (19)	0.0110 (19)
C19A	0.079 (2)	0.0738 (19)	0.0555 (19)	−0.0043 (16)	0.0164 (16)	0.0039 (16)

C20A	0.0537 (17)	0.0462 (15)	0.0496 (16)	−0.0016 (13)	0.0142 (14)	0.0028 (13)
C21A	0.062 (2)	0.079 (2)	0.0645 (19)	0.0074 (16)	0.0091 (16)	−0.0059 (16)
C22A	0.073 (2)	0.104 (3)	0.071 (2)	−0.006 (2)	0.0009 (18)	0.0038 (19)
C23A	0.104 (3)	0.085 (2)	0.062 (2)	−0.023 (2)	0.009 (2)	−0.0073 (17)
C24A	0.111 (3)	0.070 (2)	0.067 (2)	−0.009 (2)	0.036 (2)	−0.0120 (16)
C25A	0.0666 (18)	0.0615 (17)	0.0625 (18)	−0.0035 (15)	0.0256 (15)	−0.0065 (15)
N1B	0.0583 (14)	0.0602 (15)	0.0723 (16)	0.0033 (11)	0.0325 (12)	−0.0032 (12)
C2B	0.0468 (16)	0.0611 (18)	0.0580 (17)	0.0001 (13)	0.0226 (14)	−0.0036 (14)
C3B	0.083 (2)	0.0724 (19)	0.088 (2)	−0.0073 (17)	0.0549 (19)	−0.0055 (17)
C4B	0.101 (3)	0.098 (3)	0.104 (3)	−0.008 (2)	0.072 (2)	−0.008 (2)
C5B	0.085 (2)	0.084 (2)	0.102 (3)	0.0070 (19)	0.054 (2)	−0.016 (2)
C6B	0.077 (2)	0.0695 (19)	0.101 (2)	0.0023 (17)	0.053 (2)	−0.0116 (18)
N7B	0.0574 (14)	0.0529 (15)	0.0600 (14)	−0.0003 (11)	0.0322 (12)	−0.0002 (11)
C8B	0.0508 (17)	0.0536 (19)	0.0573 (18)	−0.0028 (13)	0.0174 (14)	0.0028 (14)
O9B	0.0851 (14)	0.0621 (12)	0.0752 (14)	0.0014 (10)	0.0421 (12)	0.0134 (11)
C10B	0.0479 (17)	0.0466 (15)	0.0554 (17)	0.0021 (13)	0.0210 (14)	0.0038 (13)
N11B	0.0542 (15)	0.0570 (13)	0.0637 (15)	−0.0060 (11)	0.0257 (12)	−0.0043 (11)
N12B	0.0452 (14)	0.0566 (13)	0.0564 (14)	0.0059 (11)	0.0184 (12)	−0.0021 (11)
O13B	0.0508 (11)	0.0736 (12)	0.0607 (12)	0.0089 (9)	0.0205 (9)	−0.0089 (9)
C14B	0.0406 (16)	0.0643 (18)	0.0499 (16)	0.0023 (13)	0.0106 (13)	0.0018 (13)
C15B	0.0551 (18)	0.0648 (19)	0.070 (2)	−0.0006 (15)	0.0077 (15)	−0.0024 (15)
C16B	0.061 (2)	0.088 (2)	0.077 (2)	−0.0195 (18)	0.0125 (17)	−0.0088 (17)
C17B	0.050 (2)	0.130 (3)	0.081 (2)	−0.012 (2)	0.0120 (17)	0.002 (2)
C18B	0.049 (2)	0.117 (3)	0.089 (2)	0.0193 (19)	0.0043 (17)	0.013 (2)
C19B	0.0535 (19)	0.080 (2)	0.074 (2)	0.0140 (16)	0.0120 (16)	0.0121 (16)
C25B	0.0588 (19)	0.0571 (17)	0.086 (2)	−0.0057 (15)	0.0222 (17)	−0.0033 (16)
C24B	0.065 (2)	0.063 (2)	0.123 (3)	−0.0111 (16)	0.026 (2)	−0.012 (2)
C23B	0.069 (2)	0.081 (2)	0.105 (3)	−0.0058 (18)	0.002 (2)	−0.026 (2)
C22B	0.080 (2)	0.109 (3)	0.079 (2)	−0.009 (2)	0.009 (2)	−0.010 (2)
C21B	0.065 (2)	0.081 (2)	0.074 (2)	−0.0087 (16)	0.0184 (17)	0.0061 (17)
C20B	0.0456 (16)	0.0458 (15)	0.076 (2)	0.0007 (13)	0.0191 (15)	−0.0031 (14)
O51	0.155 (5)	0.139 (4)	0.066 (3)	−0.005 (4)	0.036 (3)	0.012 (3)
C51	0.128 (7)	0.143 (8)	0.117 (8)	−0.016 (7)	0.010 (6)	0.012 (6)
O52	0.099 (11)	0.143 (11)	0.180 (14)	−0.009 (8)	0.020 (9)	0.078 (10)
C52	0.24 (2)	0.134 (16)	0.15 (2)	−0.043 (14)	0.055 (17)	−0.091 (15)

Geometric parameters (Å, º)

N1A—C2A	1.331 (3)	C3B—H3B	0.9300
N1A—C6A	1.335 (4)	C4B—C5B	1.361 (4)
C2A—C3A	1.368 (3)	C4B—H4B	0.9300
C2A—N7A	1.408 (3)	C5B—C6B	1.353 (4)
C3A—C4A	1.380 (4)	C5B—H5B	0.9300
C3A—H3A	0.9300	C6B—H6B	0.9300
C4A—C5A	1.360 (4)	N7B—C8B	1.343 (3)
C4A—H4A	0.9300	N7B—H7B	0.8600
C5A—C6A	1.366 (4)	C8B—O9B	1.214 (3)
C5A—H5A	0.9300	C8B—C10B	1.516 (4)

C6A—H6A	0.9300	C10B—N12B	1.311 (3)
N7A—C8A	1.343 (3)	C10B—N11B	1.342 (3)
N7A—H7A	0.8600	N11B—C20B	1.422 (3)
C8A—O9A	1.221 (3)	N11B—H11B	0.8600
C8A—C10A	1.511 (3)	N12B—O13B	1.344 (2)
C10A—N12A	1.315 (3)	N12B—C14B	1.438 (3)
C10A—N11A	1.347 (3)	C14B—C19B	1.363 (4)
N11A—C20A	1.417 (3)	C14B—C15B	1.381 (4)
N11A—H11A	0.8600	C15B—C16B	1.372 (4)
N12A—O13A	1.336 (2)	C15B—H15B	0.9300
N12A—C14A	1.436 (3)	C16B—C17B	1.369 (4)
C14A—C19A	1.366 (4)	C16B—H16B	0.9300
C14A—C15A	1.376 (4)	C17B—C18B	1.369 (5)
C15A—C16A	1.379 (4)	C17B—H17B	0.9300
C15A—H15A	0.9300	C18B—C19B	1.391 (4)
C16A—C17A	1.364 (5)	C18B—H18B	0.9300
C16A—H16A	0.9300	C19B—H19B	0.9300
C17A—C18A	1.365 (5)	C25B—C20B	1.374 (3)
C17A—H17A	0.9300	C25B—C24B	1.377 (4)
C18A—C19A	1.378 (4)	C25B—H25B	0.9300
C18A—H18A	0.9300	C24B—C23B	1.368 (5)
C19A—H19A	0.9300	C24B—H24B	0.9300
C20A—C21A	1.374 (4)	C23B—C22B	1.358 (5)
C20A—C25A	1.375 (4)	C23B—H23B	0.9300
C21A—C22A	1.380 (4)	C22B—C21B	1.371 (4)
C21A—H21A	0.9300	C22B—H22B	0.9300
C22A—C23A	1.358 (4)	C21B—C20B	1.3870
C22A—H22A	0.9300	C21B—H21B	0.9300
C23A—C24A	1.372 (5)	O51—C51	1.4549
C23A—H23A	0.9300	O51—H51	0.8200
C24A—C25A	1.385 (4)	C51—H51A	0.9600
C24A—H24A	0.9300	C51—H51B	0.9600
C25A—H25A	0.9300	C51—H51C	0.9600
N1B—C2B	1.332 (3)	O52—C52	1.5130
N1B—C6B	1.336 (3)	O52—H52	0.8200
C2B—C3B	1.372 (4)	C52—H52A	0.9600
C2B—N7B	1.410 (3)	C52—H52B	0.9600
C3B—C4B	1.384 (4)	C52—H52C	0.9600

C2A—N1A—C6A	116.6 (2)	C5B—C4B—C3B	119.6 (3)
N1A—C2A—C3A	123.5 (2)	C5B—C4B—H4B	120.2
N1A—C2A—N7A	112.7 (2)	C3B—C4B—H4B	120.2
C3A—C2A—N7A	123.8 (2)	C6B—C5B—C4B	118.5 (3)
C2A—C3A—C4A	118.1 (3)	C6B—C5B—H5B	120.8
C2A—C3A—H3A	121.0	C4B—C5B—H5B	120.8
C4A—C3A—H3A	121.0	N1B—C6B—C5B	124.0 (3)
C5A—C4A—C3A	119.7 (3)	N1B—C6B—H6B	118.0
C5A—C4A—H4A	120.1	C5B—C6B—H6B	118.0

C3A—C4A—H4A	120.1	C8B—N7B—C2B	126.1 (2)
C4A—C5A—C6A	117.9 (3)	C8B—N7B—H7B	116.9
C4A—C5A—H5A	121.0	C2B—N7B—H7B	116.9
C6A—C5A—H5A	121.0	O9B—C8B—N7B	126.9 (2)
N1A—C6A—C5A	124.2 (3)	O9B—C8B—C10B	119.3 (2)
N1A—C6A—H6A	117.9	N7B—C8B—C10B	113.8 (2)
C5A—C6A—H6A	117.9	N12B—C10B—N11B	118.2 (2)
C8A—N7A—C2A	126.9 (2)	N12B—C10B—C8B	118.7 (2)
C8A—N7A—H7A	116.6	N11B—C10B—C8B	123.0 (2)
C2A—N7A—H7A	116.6	C10B—N11B—C20B	127.1 (2)
O9A—C8A—N7A	126.0 (2)	C10B—N11B—H11B	116.4
O9A—C8A—C10A	119.7 (2)	C20B—N11B—H11B	116.4
N7A—C8A—C10A	114.3 (2)	C10B—N12B—O13B	119.2 (2)
N12A—C10A—N11A	117.8 (2)	C10B—N12B—C14B	124.5 (2)
N12A—C10A—C8A	119.6 (2)	O13B—N12B—C14B	116.1 (2)
N11A—C10A—C8A	122.5 (2)	C19B—C14B—C15B	121.4 (3)
C10A—N11A—C20A	127.8 (2)	C19B—C14B—N12B	120.4 (2)
C10A—N11A—H11A	116.1	C15B—C14B—N12B	118.2 (2)
C20A—N11A—H11A	116.1	C16B—C15B—C14B	119.0 (3)
C10A—N12A—O13A	119.5 (2)	C16B—C15B—H15B	120.5
C10A—N12A—C14A	123.9 (2)	C14B—C15B—H15B	120.5
O13A—N12A—C14A	116.11 (19)	C17B—C16B—C15B	120.7 (3)
C19A—C14A—C15A	121.0 (3)	C17B—C16B—H16B	119.7
C19A—C14A—N12A	120.8 (2)	C15B—C16B—H16B	119.7
C15A—C14A—N12A	118.1 (2)	C18B—C17B—C16B	119.8 (3)
C14A—C15A—C16A	119.1 (3)	C18B—C17B—H17B	120.1
C14A—C15A—H15A	120.5	C16B—C17B—H17B	120.1
C16A—C15A—H15A	120.5	C17B—C18B—C19B	120.6 (3)
C17A—C16A—C15A	120.1 (3)	C17B—C18B—H18B	119.7
C17A—C16A—H16A	120.0	C19B—C18B—H18B	119.7
C15A—C16A—H16A	120.0	C14B—C19B—C18B	118.6 (3)
C16A—C17A—C18A	120.4 (3)	C14B—C19B—H19B	120.7
C16A—C17A—H17A	119.8	C18B—C19B—H19B	120.7
C18A—C17A—H17A	119.8	C20B—C25B—C24B	119.4 (3)
C17A—C18A—C19A	120.2 (3)	C20B—C25B—H25B	120.3
C17A—C18A—H18A	119.9	C24B—C25B—H25B	120.3
C19A—C18A—H18A	119.9	C23B—C24B—C25B	120.8 (3)
C14A—C19A—C18A	119.2 (3)	C23B—C24B—H24B	119.6
C14A—C19A—H19A	120.4	C25B—C24B—H24B	119.6
C18A—C19A—H19A	120.4	C22B—C23B—C24B	119.2 (3)
C21A—C20A—C25A	119.2 (3)	C22B—C23B—H23B	120.4
C21A—C20A—N11A	122.3 (2)	C24B—C23B—H23B	120.4
C25A—C20A—N11A	118.5 (2)	C23B—C22B—C21B	121.7 (3)
C20A—C21A—C22A	120.0 (3)	C23B—C22B—H22B	119.2
C20A—C21A—H21A	120.0	C21B—C22B—H22B	119.2
C22A—C21A—H21A	120.0	C22B—C21B—C20B	118.70 (19)
C23A—C22A—C21A	121.0 (3)	C22B—C21B—H21B	120.7
C23A—C22A—H22A	119.5	C20B—C21B—H21B	120.7

C21A—C22A—H22A	119.5	C25B—C20B—C21B	120.18 (15)
C22A—C23A—C24A	119.4 (3)	C25B—C20B—N11B	117.8 (2)
C22A—C23A—H23A	120.3	C21B—C20B—N11B	121.92 (11)
C24A—C23A—H23A	120.3	C51—O51—H51	109.5
C23A—C24A—C25A	120.1 (3)	O51—C51—H51A	109.5
C23A—C24A—H24A	119.9	O51—C51—H51B	109.5
C25A—C24A—H24A	119.9	H51A—C51—H51B	109.5
C20A—C25A—C24A	120.2 (3)	O51—C51—H51C	109.5
C20A—C25A—H25A	119.9	H51A—C51—H51C	109.5
C24A—C25A—H25A	119.9	H51B—C51—H51C	109.5
C2B—N1B—C6B	116.7 (2)	C52—O52—H52	109.5
N1B—C2B—C3B	123.5 (2)	O52—C52—H52A	109.5
N1B—C2B—N7B	113.1 (2)	O52—C52—H52B	109.5
C3B—C2B—N7B	123.4 (3)	H52A—C52—H52B	109.5
C2B—C3B—C4B	117.7 (3)	O52—C52—H52C	109.5
C2B—C3B—H3B	121.2	H52A—C52—H52C	109.5
C4B—C3B—H3B	121.2	H52B—C52—H52C	109.5

C6A—N1A—C2A—C3A	−1.5 (4)	C6B—N1B—C2B—C3B	0.1 (4)
C6A—N1A—C2A—N7A	178.9 (2)	C6B—N1B—C2B—N7B	−178.8 (2)
N1A—C2A—C3A—C4A	1.4 (4)	N1B—C2B—C3B—C4B	0.2 (4)
N7A—C2A—C3A—C4A	−179.1 (2)	N7B—C2B—C3B—C4B	179.0 (3)
C2A—C3A—C4A—C5A	−0.1 (4)	C2B—C3B—C4B—C5B	0.0 (5)
C3A—C4A—C5A—C6A	−0.9 (5)	C3B—C4B—C5B—C6B	−0.4 (5)
C2A—N1A—C6A—C5A	0.4 (5)	C2B—N1B—C6B—C5B	−0.5 (5)
C4A—C5A—C6A—N1A	0.8 (5)	C4B—C5B—C6B—N1B	0.7 (5)
N1A—C2A—N7A—C8A	−169.3 (2)	N1B—C2B—N7B—C8B	−170.1 (2)
C3A—C2A—N7A—C8A	11.1 (4)	C3B—C2B—N7B—C8B	11.0 (4)
C2A—N7A—C8A—O9A	−0.2 (4)	C2B—N7B—C8B—O9B	1.5 (4)
C2A—N7A—C8A—C10A	−179.8 (2)	C2B—N7B—C8B—C10B	−179.3 (2)
O9A—C8A—C10A—N12A	61.7 (3)	O9B—C8B—C10B—N12B	−57.1 (3)
N7A—C8A—C10A—N12A	−118.6 (2)	N7B—C8B—C10B—N12B	123.6 (2)
O9A—C8A—C10A—N11A	−115.0 (3)	O9B—C8B—C10B—N11B	121.4 (3)
N7A—C8A—C10A—N11A	64.7 (3)	N7B—C8B—C10B—N11B	−57.8 (3)
N12A—C10A—N11A—C20A	−166.1 (2)	N12B—C10B—N11B—C20B	159.7 (2)
C8A—C10A—N11A—C20A	10.6 (4)	C8B—C10B—N11B—C20B	−18.9 (4)
N11A—C10A—N12A—O13A	2.6 (3)	N11B—C10B—N12B—O13B	−4.1 (3)
C8A—C10A—N12A—O13A	−174.2 (2)	C8B—C10B—N12B—O13B	174.5 (2)
N11A—C10A—N12A—C14A	−169.2 (2)	N11B—C10B—N12B—C14B	171.1 (2)
C8A—C10A—N12A—C14A	13.9 (3)	C8B—C10B—N12B—C14B	−10.3 (4)
C10A—N12A—C14A—C19A	51.8 (3)	C10B—N12B—C14B—C19B	−56.8 (4)
O13A—N12A—C14A—C19A	−120.2 (3)	O13B—N12B—C14B—C19B	118.6 (3)
C10A—N12A—C14A—C15A	−131.3 (3)	C10B—N12B—C14B—C15B	126.2 (3)
O13A—N12A—C14A—C15A	56.6 (3)	O13B—N12B—C14B—C15B	−58.4 (3)
C19A—C14A—C15A—C16A	−2.0 (4)	C19B—C14B—C15B—C16B	1.6 (4)
N12A—C14A—C15A—C16A	−178.8 (2)	N12B—C14B—C15B—C16B	178.6 (2)
C14A—C15A—C16A—C17A	0.3 (4)	C14B—C15B—C16B—C17B	−1.4 (4)
C15A—C16A—C17A—C18A	1.2 (5)	C15B—C16B—C17B—C18B	0.2 (5)

C16A—C17A—C18A—C19A	−1.0 (5)	C16B—C17B—C18B—C19B	0.7 (5)
C15A—C14A—C19A—C18A	2.2 (4)	C15B—C14B—C19B—C18B	−0.7 (4)
N12A—C14A—C19A—C18A	179.0 (3)	N12B—C14B—C19B—C18B	−177.6 (3)
C17A—C18A—C19A—C14A	−0.7 (5)	C17B—C18B—C19B—C14B	−0.5 (5)
C10A—N11A—C20A—C21A	35.0 (4)	C20B—C25B—C24B—C23B	2.7 (4)
C10A—N11A—C20A—C25A	−147.9 (2)	C25B—C24B—C23B—C22B	−1.4 (5)
C25A—C20A—C21A—C22A	−0.3 (4)	C24B—C23B—C22B—C21B	−0.8 (5)
N11A—C20A—C21A—C22A	176.7 (2)	C23B—C22B—C21B—C20B	1.6 (4)
C20A—C21A—C22A—C23A	−1.7 (5)	C24B—C25B—C20B—C21B	−1.8 (3)
C21A—C22A—C23A—C24A	1.7 (5)	C24B—C25B—C20B—N11B	174.1 (2)
C22A—C23A—C24A—C25A	0.2 (5)	C22B—C21B—C20B—C25B	−0.3 (2)
C21A—C20A—C25A—C24A	2.3 (4)	C22B—C21B—C20B—N11B	−176.0 (3)
N11A—C20A—C25A—C24A	−174.9 (2)	C10B—N11B—C20B—C25B	152.2 (2)
C23A—C24A—C25A—C20A	−2.3 (4)	C10B—N11B—C20B—C21B	−32.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7*A*—H7*A*···O13*B*	0.86	1.94	2.730 (3)	152
N11*A*—H11*A*···N1*B*^{i}	0.86	2.30	3.095 (3)	153
N11*A*—H11*A*···O13*A*	0.86	2.25	2.603 (3)	105
N7*B*—H7*B*···O13*A*^{ii}	0.86	1.90	2.724 (3)	160
N11*B*—H11*B*···N1*A*	0.86	2.32	3.056 (3)	144
N11*B*—H11*B*···O13*B*	0.86	2.28	2.604 (3)	102
O51—H51···O13*B*	0.82	1.95	2.770 (3)	179

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*, *y*−1, *z*.